(19)日本国特許庁（ＪＰ）　(12) 公開特許公報（Ａ）　(11)特許出願公開番号
特開2003－128466
（P2003－128466A）
(43)公開日　平成15年5月8日(2003.5.8)

(51)Int.Cl.7	識別記号	FI	テーマコード（参考）
C04B 35/58	105	C04B 35/58	105A 4G001
// B23C 5/16		B23C 5/16	

審査請求 未請求 請求項の数10 OL （全 6 頁）

(21)出願番号　特願2001－322752(P2001－322752)

(22)出願日　平成13年10月19日(2001.10.19)

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(54)【発明の名称】　硼化物焼結体とその製造方法

(57)【要約】
【課題】　鋼の切削などに必要な耐欠損性を具える硼化物焼結体を提供する。
【解決手段】　Alと周期律表IaおよびIIa族から選ばれる一つの元素Mと硼素とからなる硼化物を含む硼化物焼結体である。この硼化物焼結体は、組成が$Al_{x \leq 1}M_{y \leq 1}B_{z \geq 12}$で表される硼化物と不可避的不純物からなる。そして、この焼結体に占める硼化物の含有量が98体積%以上である。この焼結体は、実質的に硼化物だけからなる焼結体として構成されるため、切削工具として用いた場合に優れた耐摩耗性と耐欠損性とを具える。

【特許請求の範囲】

【請求項1】 Alと周期律表IaおよびIIa族から選ばれる一つの元素Mと硼素とからなる硼化物を含む硼化物焼結体であって、

前記硼化物焼結体は、組成が$Al_{X\leq 1}M_{y\leq 1}B_{Z\geq 12}$で表される硼化物と不可避的不純物からなり、

この焼結体に占める前記硼化物の含有量が98体積%以上であることを特徴とする硼化物焼結体。

【請求項2】 焼結体を構成する硼化物の最大粒径が3μm以下で、硼化物の平均粒径が1μm以下であることを特徴とする請求項1に記載の硼化物焼結体。

【請求項3】 硼化物の硬度がHv25GPa以上であることを特徴とする請求項1に記載の硼化物焼結体。

【請求項4】 前記元素MがMgであることを特徴とする請求項1に記載の硼化物焼結体。

【請求項5】 前記元素MがLiであることを特徴とする請求項1に記載の硼化物焼結体。

【請求項6】 最大粒径が3μm以下の硼化物粒子を圧力：150MPa以上10GPa以下、温度：1000℃以上1500℃以下で焼結する工程を具えることを特徴とする硼化物焼結体の製造方法。

【請求項7】 最大粒径が3μm以下の硼化物粒子は、硼素：最大粒径1μm以下で75〜91原子%、Al：4〜21原子%、周期律表IaおよびIIa族から選ばれる一つの元素M：3〜6原子%の組成を有する原料を、窒素以外の不活性ガス雰囲気中にて、1300℃以下の温度で加熱処理する工程により得ることを特徴とする請求項6に記載の硼化物焼結体の製造方法。

【請求項8】 最大粒径が5μm以下の硼化物粒子を圧力：150MPa以上10GPa以下、温度：1000℃以上1500℃以下で焼結する工程を具えることを特徴とする硼化物焼結体の製造方法。

【請求項9】 最大粒径が5μm以下の硼化物粒子は、次の工程により得ることを特徴とする請求項８に記載の硼化物焼結体の製造方法。

①硼素：最大粒径40μm以下で75〜91原子%、Al：4〜21原子%、周期律表IaおよびIIa族金属から選ばれる一つの元素M：3〜6原子%の組成を有する原料を、窒素以外の不活性ガス雰囲気中にて、1300℃以下の温度で加熱処理し、最大粒径が50μm以下で、かつ平均粒径が15μm以下の硼化物粒子を得る工程

②この最大粒径が50μm以下の硼化物粒子を粉砕する工程

【請求項10】 焼結圧力を1GPa以上5GPa以下としたことを特徴とする請求項６または８に記載の硼化物焼結体の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、耐摩耗材料や切削工具に好適な硼化物焼結体とその製造方法に関するものである。特に、鋼や鋳鉄の加工耐摩工具や切削工具に最適な高硬度の硼化物焼結体とその製造方法に関するものである。

【0002】

【従来の技術】切削用のAl_2O_3やSi_3N_4セラミック工具は耐摩耗性に優れるが靭性に乏しく、断続切削では十分な工具寿命を得られない。一方、cBN焼結体はより長寿命を示すが、超高圧下で合成されるcBN粉末原料が高価である。さらにcBN焼結体はcBNの安定領域である4GPa以上の超高圧で焼結するため、工具単価が高く、長寿命であっても、コストメリットを得られない場合がある。

【0003】これに対し、$AlMgB_{14}$や$AlLiB_{14}$の単結晶はHv3000程度の高硬度を示し、頑強な結晶構造は耐欠損性と耐摩耗性を兼備した工具素材として期待できる。

【0004】$AlMgB_{14}$に関する従来技術として、W.Higashi and T.Ito J.Less Comm.Met.92 (1983) 239に記載の技術が知られている。この論文は、多量のアルミニウム融液中にマグネシウムと硼素を溶解し、結晶を析出させる方法を開示している。

【0005】また、別の従来技術としてUSP6099605に記載の技術が知られている。この公報は、Al、Mg、B元素の粉末をメカニカルアロイング法を用いて微細化した後にホットプレス装置で焼結することにより、高硬度の焼結体が得られることを示している。

【0006】

【発明が解決しようとする課題】前記論文の方法では数十μm以上の$AlMgB_{14}$粒子が生成できる。より具体的には、粒径が最小でも50μmであり、最大で2mmに達する。しかし、このような粗粒では緻密な焼結体を得ることができないという問題があった。また、このような粗粒を粉砕した場合、微細化に長時間を要し、粉砕容器や粉砕媒体（ボールなど）から不純物が3重量%以上と多量に混入し、耐摩耗性や耐欠損性を評価し得る焼結体は得られていない。

【0007】一方、USP6099605に記載の技術では、鋼製の粉砕ボールと原料粉末を入れた高エネルギータイプの粉砕装置でメカニカルアロイング過程を行うため、粉砕ボールや容器材質の不純物混入が避けられない。そのため、鉄と原料硼素が反応してFeB_{49}が生成し混入していることが確認されている。このような金属の2元硼化物は脆性材料であり、焼結体を製作して切削工具として用いた場合に刃先の耐欠損性を低下させると言う問題がある。また、メカニカルアロイングでは酸化反応が生じやすく、さらに急激な酸化反応による爆発の恐れがあるために、LiやNa等のIa、IIa族金属を含む硼化物を微細化して焼結体を得ることは実質的に不可能である。

【0008】従って、本発明の主目的は、鋼の切削などに必要な耐欠損性を具える硼化物焼結体とその製造方法とを提供することにある。また、本発明の他の目的は、低コストで密度の高い硼化物焼結体とその製造方法とを

提供することにある。

【0009】

【課題を解決するための手段】本発明は、組成や原料の粒径あるいは原料粉末の合成条件を規定することで上記の目的を達成する。すなわち、本発明硼化物焼結体は、Alと周期律表IaおよびIIa族金属から選ばれる一つの元素Mと硼素とからなる硼化物を含む硼化物焼結体である。この硼化物焼結体は、組成が$Al_{X\leqq 1}M_{y\leqq 1}B_{Z\geqq 12}$で表される硼化物と不可避的不純物からなる。そして、この焼結体に占める硼化物の含有量が98体積%以上であることを特徴とする。

【0010】従来、組成が$Al_{X\leqq 1}M_{y\leqq 1}B_{Z\geqq 12}$で表される硼化物を含む焼結体は、硼化物の含有量が低く、例えば、USP6099605に記載の技術では10重量%のオーダーで不純物が含まれている。これは、メカニカルアロイングで結晶構造をほぼ完全に破壊してしまうような粉砕を原料粉末に対して行っているため、その際に不純物が混入するからである。本発明では、結晶構造の極力崩れていない微細な粉末を原料に用いることで、硼化物の含有量の高い焼結体を得ることができ、切削工具に最適な高硬度と耐欠損性とを兼備することができる。

【0011】本発明焼結体は、組成が$Al_{X\leqq 1}M_{y\leqq 1}B_{Z\geqq 12}$で表される三元硼化物と、２体積%未満の不可避的不純物とからなる。より具体的な硼化物の組成としては、$AlMgB_{14}$や$AlLiB_{14}$などが挙げられる。$AlMgB_{14}$の場合、より正確な構造は$Al_{0.75}Mg_{0.78}B_{14}$である。不可避的不純物としては、一般にAl-Mg-B系ではAlおよびMgを含む酸化物が見られ、Al-Li-B系ではAlおよびLiを含む酸化物が見られる。AlおよびMgを含む酸化物としては、$MgAl_2O_4$が、AlおよびLiを含む酸化物としては$AlLiO_2$が挙げられる。

【0012】焼結体を構成する硼化物の最大粒径を3μm以下とし、硼化物の平均粒径を1μm以下とすることが好ましい。このような微細な硼化物を構成することにより、緻密で高硬度の焼結体を得ることができる。切削工具として好ましい焼結体の硬度はHv25GPa以上である。また、焼結体の理論密度に対する相対密度は97%以上が好ましい。

【0013】一方、本発明硼化物焼結体の製造方法は、最大粒径が3μm以下または5μm以下の硼化物粒子を圧力：150MPa以上10GPa以下、温度：1000℃以上1500℃以下で焼結する工程とを具えることを特徴とする。

【0014】微細な硼化物粒子を用いることで、緻密で高硬度の焼結体を得ることができる。このような硼化物粒子は、粉砕を行わないか短時間の粉砕とすることで結晶構造を極力崩さず、かつ不純物の少ない状態とすることができ、硼化物含有量の高い焼結体を作製する際の原料粉末として好適である。焼結圧力のより好ましい範囲は、1GPa以上5GPa以下、更に好ましくは、1GPa以上3GPa以下である。焼結温度のより好ましい範囲は1200〜1400℃である。焼結保持時間は、15〜60分程度が好ましい。

【0015】焼結体の原料粉末となる微細な硼化物粒子は、粉砕を行うことなく当初から微粒の硼化物粒子を得る方法と、まず粗粒を得て、その後粉砕することで微粒とする方法の2通りがある。

【0016】粉砕を行わない方法は、硼素：最大粒径1μm以下で75〜91原子%、Al：4〜21原子%、周期律表IaおよびIIa族から選ばれる一つの元素M：3〜6原子%の組成を有する原料を、窒素以外の不活性ガス雰囲気中にて、1300℃以下の温度で加熱処理する工程により得ることができる。

【0017】最大粒径が1μm以下の硼素を原料とすることで、得られる硼化物粒子の最大粒径を3μm以下に抑えることができる。

【0018】従来、$AlMgB_{14}$では、結晶成長させるためにAlが多く含まれていた。例えばAlの含有量は80〜91原子%程度である。本発明では、極力Alの含有量を減らし、微細な硼化物粒子が得られる組成を選択した。上記に規定した組成範囲を外れると、結晶粒成長が起こり、粗粒の硼化物粒子となる傾向がある。

【0019】$AlMgB_{14}$について、Alの含有量を変化させて上記の方法にて微粒の硼化物粒子が得られた組成例を表1に示す。この表1における組成No.1は$AlMgB_{14}$の化学量論組成で、微細な硼化物粒子が得られたが、若干の酸化物も見られた。また、No.2は得られた粒子が微粒であり、酸化物も非常に少なく好ましい$AlMgB_{14}$であった。さらに、No.3でも微粒の$AlMgB_{14}$が合成できたが、No.2に比べて粗粒化しており、好ましい微粒子が得られるAl量としては21原子%と思われる。

【0020】

【表1】

No.	at%				wt%（左記at%の換算）			
	B	Al	Mg	合計	B	Al	Mg	合計
1	90.15	4.83	5.02	100	79.43	10.62	9.95	100
2	86.00	9.21	4.79	100	71.81	19.19	8.99	100
3	75.55	20.24	4.21	100	55.74	37.27	6.99	100

【0021】加熱処理の雰囲気は、不活性ガス雰囲気とする。ただし、窒素は除く。通常、アルゴンを用いることが好適である。

【0022】加熱処理温度は、理論上、Alの融点（660.4℃）以上の温度〜1300℃以下の温度であれば良い。1300℃を超える温度で合成した場合はAlB_{12}も同時に合成し

てしまうため好ましくない。最適な合成温度は1200～1300℃、特に1200℃前後である。保持時間は、30～90分程度が好ましい。

【0023】この方法であれば、粉砕を行うことなく微粒の硼化物粒子を得ることができ、粉砕時に不純物が混入して焼結体とした際に硼化物の含有量が低下することを回避できる。

【0024】次に、粉砕を行う方法は、まず硼素：最大粒径40μm以下で75～91原子%、Al：4～21原子%、周期律表IaおよびIIa族から選ばれる一つの元素M：3～6原子%の組成を有する原料を、窒素以外の不活性ガス雰囲気中にて、1300℃以下の温度で加熱処理し、最大粒径が50μm以下で、かつ平均粒径15μm以下の硼化物粒子を得る。続いて、この最大粒径が50μm以下の硼化物粒子を粉砕して最大粒径5μm以下の硼化物粒子とする。

【0025】原料の組成、加熱処理における雰囲気、温度は粉砕を行わない方法の場合と同様である。ここでは原料となる硼素の粒径が粗いため合成された硼化物粒径も大きくなるが、最大粒径が50μm以下で、平均粒径を15μm以下とすることで、粉砕工程を用いても短時間で最大粒径5μm以下に微細化できる。粉砕時間が短かければ、不純物の混入がほとんど無い状態で硼化物粉末を得ることができる。そして、不純物の少ない硼化物粒子を用いることで焼結性も改善され、緻密な焼結体を得ることができる。

【0026】粉砕は、極力不純物が混入しないような条件を選択して行うことが好ましい。ボールミルを用いた粉砕条件例としては、ボール：アルミナ製、直径3～6mm、粉砕時間：4～10時間が挙げられる。できるだけ、短時間で粉砕を行うことが好ましい。

【0027】以上のように合成された微細な硼化物粒子は、TiやTiC、AlN、Al_2O_3等を結合材として用いて焼結体を得ることも可能である。

【0028】

【発明の実施の形態】以下、本発明の実施の形態を説明する。

（実施例1）純度99.9%、最大粒径1μm以下の無定形硼素粉末、純度99.9%、最大粒径40μm以下のアルミニウム粉末、純度99.9%、最大粒径180μmのマグネシウム粉末をそれぞれ85原子%、10原子%、5原子%配合混合した。この混合粉末を高純度なアルミナるつぼに入れ、高温雰囲気加熱炉中に設置し、99.99%以上の高純度アルゴンガス雰囲気中、1200℃で1時間保持したのち、アルゴンガス雰囲気を保持したまま冷却した。加熱後の粉末を塩酸中で溶解処理して余分のAlを除去した。残留した粉末をX線回折測定で調べたところ、$AlMgB_{14}$のみの回折ピークを示した。粒度測定を行なったところ、最大で2μm、平均で0.7μmの粒径からなる微細粉末であった。

【0029】この粉末を10^{-5}torr（1.33×10^{-6}kPa）の真空中、600℃で乾燥処理したのち、ペレット状に加圧成形した。この成形体をホットプレス装置を用いて圧力180MPa、温度1300℃で1時間保持して焼結体を得た。得られた焼結体は理論密度の2.6g/cm³に対する相対密度が98.6%で、ビッカース硬度を測定したところ、27GPaであった。さらに焼結体をX線回折測定によって分析したところ、$AlMgB_{14}$の他に、わずかに酸化物である$MgAl_2O_4$の回折ピークも観察された。組成が既知である混合物のX線回折測定から得られたピーク強度比の検量線より、この焼結体中で$AlMgB_{14}$は98.4体積%の含有率であった。

【0030】（実施例2）実施例1と同様に$AlMgB_{14}$の粉末を作製し、この粉末を10^{-5}torr（1.33×10^{-6}kPa）の真空中、600℃で乾燥処理したのち、ペレット状に加圧成形した。この成形体を超高圧高温装置を用いて圧力2GPa、温度1300℃で30分間保持して焼結体を得た。得られた焼結体は理論密度の2.6g/cm³に対する相対密度が99.7%の緻密な組織を有し、ビッカース硬度を測定したところ、32GPaであった。実施例1と同様にX線回折測定を行った結果、$AlMgB_{14}$の含有率は99.5体積%であった。

【0031】（実施例3）純度99.9%、最大粒径40μm以下の結晶質（三方晶系）硼素粉末、純度99.9%、最大粒径40μm以下のアルミニウム粉末、純度99.9%、最大粒径180μmのマグネシウム粉末をそれぞれ85原子%、10原子%、5原子%配合混合した。この混合粉末を高純度なアルミナるつぼに入れ、高温雰囲気加熱炉中に設置し、99.99%以上の高純度アルゴンガス雰囲気中、1200℃で1時間保持したのち、アルゴンガス雰囲気を保持したまま、冷却した。加熱後の粉末を塩酸中で溶解処理した。残留した粉末をX線回折測定で調べたところ、$AlMgB_{14}$のみの回折ピークを示した。粒度測定を行なったところ、最大粒径が50μm以下で、平均粒径で15μmであった。

【0032】この粉末をボールミルを用いて、エタノール中で最大粒径が5μm以下になるまで粉砕した。粉砕後の粉末を10^{-5}torr（1.33×10^{-6}kPa）の真空中、600℃で乾燥処理したのち、ペレット状に加圧成形した。この成形体をホットプレス装置を用いて圧力180MPa、温度1200℃で1時間保持して焼結した。得られた焼結体は理論密度の2.6g/cm³に対する相対密度97%で、ビッカース硬度は25.5GPaであった。実施例1と同様にX線回折測定を行った結果、$AlMgB_{14}$の含有率は98.3体積%であった。

【0033】（実施例4）実施例3と同様にして、$AlMgB_{14}$の粉砕粉末を作製し、この粉末を10^{-5}torr（1.33×10^{-6}kPa）の真空中、600℃で乾燥処理したのち、ペレット状に加圧成形した。この成形体を超高圧高温装置を用いて圧力2GPa、温度1300℃で30分間保持して焼結体を得た。得られた焼結体は相対密度が99.5%の緻密な組織を有し、ビッカース硬度を測定したところ、32.3GPaであった。実施例1と同様にX線回折測定を行った結果、$AlMgB_{14}$の含有率は99.0体積%であった。

【0034】（実施例5）硼素とアルミニウムとリチウ

ムの組成比が82原子%、13原子%、5原子%となるように、「純度99.9%、最大粒径1μm以下の無定形硼素粉末」と、「純度99.9%、最大粒径300μm以下のアルミニウム−28原子%リチウム合金粉末」とをそれぞれ70重量%と30重量%配合混合した。この混合粉末を高純度なアルミナるつぼに入れ、高温雰囲気加熱炉中に設置し、99.99%以上の高純度アルゴンガス雰囲気中、1200℃で1時間保持したのち、アルゴンガス雰囲気を保持したまゝ冷却した。加熱後の粉末を塩酸中で溶解処理した。残留した粉末をX線回折測定で調べたところ、$AlLiB_{14}$のみの回折ピークを示した。粒度測定を行ったところ、最大で3μm、平均で0.8μmの粒径からなる微細粉末であった。

【0035】この粉末を10^{-5}torr（1.33×10^{-5}kPa）の真空中、600℃で乾燥処理したのち、ペレット状に加圧成形した。この成形体をホットプレス装置を用いて圧力180MPa、温度1300℃で1時間保持して焼結体を得た。得られた焼結体は理論密度の2.46g/cm³に対する相対密度98%で、ビッカース硬度を測定したところ、30GPaであった。実施例1と同様にX線回折測定を行った結果、酸化物として$AlLiO_2$の回折ピークが観察され、焼結体中の$AlLiB_{14}$の含有率は99.6%であった。

【0036】（試験例1）実施例1〜5の焼結体を超硬合金の台金上に接合し、これを切れ刃とした切削工具を作製し、熱処理したS45C（H_RC35）の丸棒（φ150×300mm）の外周切削を10分間行なった。切削条件はV=300m/min、d=0.5mm、f=0.15mm/rev.、乾式である。

【0037】比較例1として、実施例3の方法で作製した最大粒径が50μm以下で、平均粒径で15μmの粒子をホットプレスで圧力180MPa、温度1300℃で1時間保持して得られた焼結体から切削工具を準備した。さらに、比較例2として市販のアルミナ系セラミック工具、比較例3として市販のサーメット工具も用いた。その結果を表2に示す。

【0038】

【表2】

	切削時間	逃げ面摩耗量
実施例1	10分	0.179mm
実施例2	10分	0.140mm
実施例3	10分	0.154mm
実施例4	10分	0.113mm
実施例5	10分	0.203mm
比較例1	2分欠損	−
比較例2	10分	0.328mm
比較例3	欠損	−

【0039】表2に示すように、実施例は優れた耐摩耗性を示し、長寿命を示した。一方、粗粒のままの焼結体は強度が低く、切削途中に欠損した。アルミナ系工具は加工途中で欠損により短寿命を示した。サーメット工具は靭性が低く、切削開始と同時に刃先が大破した。

【0040】（試験例2）試験例1と同様に実施例と比較例の切削工具を作製し、熱処理したSCM435（H_RC40）の丸棒（φ150×300mm）に4本のV字溝を入れた被削材を準備し、工具に衝撃が負荷される断続切削を行なった。切削条件はV=200m/min、d=0.2mm、f=0.12mm/rev.、乾式である。その結果を表3に示す。

【0041】

【表3】

	欠損寿命
実施例1	18分
実施例2	23分
実施例3	10分
実施例4	10分
実施例5	19分
比較例1	0.1分
比較例2	2分
比較例3	1分

【0042】表3に示すように、実施例は優れた耐欠損性を示した。一方、粗粒のままの焼結体とアルミナ系工具は強度が不足したために切削初期に欠損した。サーメット工具は熱亀裂が発生し欠損した。

【0043】

【発明の効果】以上説明したように、本発明焼結体は硼化物の含有量が高く、実質的に硼化物だけからなる焼結体として構成され、切削工具として用いた場合に優れた耐摩耗性と耐欠損性とを備える。

【0044】また、本発明焼結体の製造方法は、微粒の原料粉末を用いることで緻密で高硬度の焼結体を得ることができる。

フロントページの続き

Fターム(参考) 4G001 BA61 BA63 BA68 BB41 BC13
BC42 BC52 BC54 BC55 BD12
BD18 BE21

CLAIMS

[Claim 1] It is the boride sintered compact which is a boride sintered compact containing the boride which consists of one element M chosen from aluminum, a periodic table Ia, and an IIa group, and boron, and is characterized by the content of the aforementioned boride which the aforementioned boride sintered compact consists of the boride and unescapable impurity by which composition is expressed with AlX<=1My<=1BZ>=12, and is occupied to this sintered compact being more than 98 volume %.

[Claim 2] The boride sintered compact according to claim 1 with which the maximum droplet size of the boride which constitutes a sintered compact is characterized by the mean particle diameter of a boride being 1 micrometer or less by 3 micrometers or less.

[Claim 3] The boride sintered compact according to claim 1 characterized by the degree of hardness of a boride being more than Hv25GPa.

[Claim 4] The boride sintered compact according to claim 1 characterized by the aforementioned element M being Mg.

[Claim 5] The boride sintered compact according to claim 1 characterized by the aforementioned element M being Li.

[Claim 6] a maximum droplet size -- a boride particle 3 micrometers or less -- pressure: -- the manufacture method of the boride sintered compact characterized by having 150 or more-MPa 10 or less GPa, and the process sintered less than [more than temperature:1000 degree-C1500 degree C]

[Claim 7] For a boride particle 3 micrometers or less, a maximum droplet size is the manufacture method of a boride sintered compact according to claim 6 that it is characterized by obtaining according to the process at which the raw material with which it has composition of one element M:3 with which it is chosen out of 75 to 91 atom %, four to aluminum:21 atom %, a periodic table Ia, and an IIa group by 1 micrometer or less of boron:maximum droplet sizes - 6 atom % is heat-treated at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen.

[Claim 8] a maximum droplet size -- a boride particle 5 micrometers or less -- pressure: -- the manufacture method of the boride sintered compact characterized by having 150 or more-MPa 10 or less GPa, and the process sintered less than [more than temperature:1000 degree-C1500 degree C]

[Claim 9] For a boride particle 5 micrometers or less, a maximum droplet size is the manufacture method of a boride sintered compact according to claim 8 that it is characterized by obtaining according to the following process.

By 40 micrometers or less of maximum droplet sizes Boron : ** 75 to 91 atom %, four to aluminum:21 atom %, The raw material which has composition of one element M:3 chosen from a periodic table Ia and an IIa group metal - 6 atom % is heat-treated at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen. a maximum droplet size by 50 micrometers or less and process ** from which a mean particle diameter obtains a boride particle 15 micrometers or less -- the process at which this maximum droplet size grinds a boride particle 50 micrometers or less [Claim 10] The manufacture method of the boride sintered compact according to claim 6 or 8 characterized by setting a sintering pressure to 5 or less GPa more than per GPa.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] this invention relates to the suitable boride sintered compact for antifriction material or a cutting tool, and its manufacture method. Especially, it is related with the optimal boride sintered compact and its optimal manufacture method of a high degree of hardness for a processing abrasion-resistant tool or a cutting tool of steel or cast iron.
[0002]
[Description of the Prior Art] Although aluminum 2O3 for cutting and Si3N4 ceramic tool are excellent in abrasion resistance, they are lacking in toughness, and they cannot acquire tool life sufficient in intermittent cutting. On the other hand, although a cBN sintered compact shows a longevity life more, the cBN powder raw material compounded under extra-high voltage is expensive. Furthermore, since a cBN sintered compact sinters by the extra-high voltage of 4 or more GPas which is the stable zone of cBN, even if a tool unit price is high and it long lasting, a cost merit cannot be obtained. [unit price]
[0003] On the other hand, the single crystal of AlMgB14 or AlLiB14 shows an about 3000-Hv high degree of hardness, and the dogged crystal structure can be expected as a tool material which combined deficit-proof nature and abrasion resistance.
[0004] As conventional technology about AlMgB14, it is W.Higashi. and T.Ito J.Less The technology of a publication is known by Comm.Met.92 (1983) 239. This paper dissolves magnesium and boron into a lot of aluminum melts, and the method of depositing a crystal is indicated.
[0005] Moreover, the technology of a publication is known by USP6099605 as another conventional technology. This official report shows that the sintered compact of a high degree of hardness is obtained by sintering the powder of aluminum, Mg, and B element with hotpress equipment, after turning minutely using the mechanical alloying method.
[0006]
[Problem(s) to be Solved by the Invention] By the method of the aforementioned paper, AlMgB14 particle dozens of micrometers or more is generable. More specifically, the minimum is also 50 micrometers and particle size amounts to 2mm at the maximum. However, there was a problem that a precise sintered compact could not be obtained, in such coarse grain. Moreover, when such coarse grain is ground, detailed-ization takes a long time, an impurity mixes in 3 % of the weight or more and a large quantity from a pulverization container or tumbling medias (ball etc.), and the sintered compact which can evaluate abrasion resistance and deficit-proof nature is not obtained.
[0007] On the other hand, in order for the pulverization equipment of the high-energy type into which a steel crushing steel ball and steel raw material powder were put to perform mechanical alloying process to USP6099605 with the technology of a publication, impurity mixing of a crushing steel ball or the container quality of the material is not avoided. Therefore, it is checked that iron and raw material boron react, and FeB49 is generating and mixing. The 2 yuan boride of such a metal is a brittle material, and when a sintered compact is manufactured and it uses as a cutting tool, there is a problem referred to as reducing the deficit-proof nature of the edge of a blade.

Moreover, since there is fear of explosion by still more rapid oxidation reaction, it is [that it is easy to produce oxidation reaction in mechanical alloying] substantially impossible to turn minutely the boride containing Ia(s), such as Li and Na, and an IIa group metal, and to obtain a sintered compact.

[0008] Therefore, the key objective of this invention is to offer a boride sintered compact equipped with deficit-proof nature required for cutting of steel etc., and its manufacture method. Moreover, other purposes of this invention are to offer the high boride sintered compact and its manufacture method of density by the low cost.

[0009]

[Means for Solving the Problem] this invention attains the above-mentioned purpose by specifying composition, the particle size of a raw material, or the synthetic conditions of raw material powder. That is, this invention boride sintered compact is a boride sintered compact containing the boride which consists of one element M chosen from aluminum, a periodic table Ia, and an IIa group metal, and boron. This boride sintered compact consists of the boride and unescapable impurity by which composition is expressed with AlX<=1My<=1BZ>=12. And it is characterized by the content of the boride occupied to this sintered compact being more than 98 volume %.

[0010] Conventionally, the sintered compact containing the boride to which composition is expressed with AlX<=1My<=1BZ>=12 has the low content of a boride, for example, the impurity is contained in USP6099605 to 10% of the weight of order with the technology of a publication. This is because an impurity mixes in that case since pulverization which destroys the crystal structure nearly completely by mechanical alloying is performed to raw material powder. In this invention, by using for a raw material the detailed powder with which the crystal structure has not collapsed as much as possible, the high sintered compact of the content of a boride can be obtained and the optimal high degree of hardness for a cutting tool and deficit-proof nature can be combined.

[0011] this invention sintered compact consists of a 3 yuan boride to which composition is expressed with AlX<=1My<=1BZ>=12, and an unescapable impurity of under 2 volume %. AlMgB14, AlLiB14, etc. are mentioned as composition of a more concrete boride. In AlMgB14, more exact structure is aluminum0.75Mg 0.78B14. As an unescapable impurity, generally, by the aluminum-Mg-B system, the oxide containing aluminum and Mg is seen and the oxide containing aluminum and Li is seen by the aluminum-Li-B system. As an oxide containing aluminum and Mg, AlLiO2 is mentioned as an oxide with which MgAl 2O4 contains aluminum and Li.

[0012] It is desirable to set to 3 micrometers or less the maximum droplet size of the boride which constitutes a sintered compact, and to set the mean particle diameter of a boride to 1 micrometer or less. By constituting such a detailed boride, the precise sintered compact of a high degree of hardness can be obtained. The degree of hardness of a sintered compact desirable as a cutting tool is more than Hv25GPa. Moreover, 97% or more of the relative density to the theoretical density of a sintered compact is desirable.

[0013] On the other hand, the manufacture method of this invention boride sintered compact is characterized by having the process at which a maximum droplet size sinters a boride particle (3 micrometers or less or 5 micrometers or less) less than [more than pressure:150MPa10GPa] and less than [more than temperature:1000 degree-C1500 degree C].

[0014] By using a detailed boride particle, the precise sintered compact of a high degree of hardness can be obtained. Such a boride particle is suitable as raw material powder at the time of being unable to break down the crystal structure as much as possible because it considers [whether it grinds and] as short-time pulverization, and being able to consider as a state with few impurities, and producing the high sintered compact of a boride content. The more desirable range of 1 or more-GPa 5 or less GPa of a sintering pressure is 3 or less GPa more than per GPa still more preferably. The more desirable range of sintering temperature is 1200-1400 degrees C. The sintering holding time has about 15 - 60 desirable minutes.
[0015] The detailed boride particle used as the raw material powder of a sintered compact has two kinds, the method of obtaining the boride particle of a particle from the beginning, without grinding, and the method of making it into a particle by obtaining coarse grain first and grinding after that.
[0016] The method which does not grind can be acquired according to the process which heat-treats the raw material which has composition of one element M:3 chosen from 75 to 91 atom %, four to aluminum:21 atom %, a periodic table Ia, and an IIa group by 1 micrometer or less of boron:maximum droplet sizes - 6 atom % at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen.
[0017] The maximum droplet size of the boride particle obtained can be held down to 3 micrometers or less because a maximum droplet size uses boron 1 micrometer or less as a raw material.
[0018] Conventionally, in $AlMgB_{14}$, in order to carry out a crystal growth, many aluminum was contained. For example, the content of aluminum is a 80 - 91 atom % grade. In this invention, the content of aluminum was reduced as much as possible, and the composition from which a detailed boride particle is obtained was chosen. When it separates from the composition range specified above, a grain growth happens and there is an inclination used as the boride particle of coarse grain.
[0019] The example of composition from which the content of aluminum was changed and the boride particle of a particle was obtained by the above-mentioned method about $AlMgB_{14}$ is shown in Table 1. Some oxide was also seen, although composition No.1 in this table 1 is the stoichiometric composition of $AlMgB_{14}$ and the detailed boride particle was obtained. Moreover, the obtained particle was a particle and No.2 were desirable $AlMgB_{14}$ also with very little oxide. Furthermore, although No.3 have compounded $AlMgB_{14}$ of a particle, it has coarse-grain-ized compared with No.2, and 21 atom % is thought as an amount of aluminum from which a desirable particle is obtained.
[0020]
[Table 1]

No	at%				wt% (左記at%の換算)			
	B	Al	Mg	合計	B	Al	Mg	合計
1	90.15	4.83	5.02	100	79.43	10.62	9.95	100
2	86.00	9.21	4.79	100	71.81	19.19	8.99	100
3	75.55	20.24	4.21	100	55.74	37.27	6.99	100

[0021] Atmosphere of heat-treatment is made into inert gas atmosphere. However, nitrogen is removed. Usually, it is suitable to use an argon.
[0022] Heat-treatment temperature should just be the temperature not more than temperature -1300 degree C beyond the melting point (660.4 degrees C) of aluminum

theoretically. It is not desirable in order to compound AlB12 simultaneously, when it compounds at the temperature exceeding 1300 degrees C. 1200-1300 degrees C especially of optimal synthetic temperature are before and after 1200 degrees C. About 30 - 90 minutes of the holding time are desirable.

[0023] When it was this method, and the boride particle of a particle can be obtained, an impurity mixes at the time of pulverization and it considers as a sintered compact, without grinding, it can avoid that the content of a boride falls.

[0024] Next, the method of grinding heat-treats the raw material which has composition of one element M:3 first chosen from 75 to 91 atom %, four to aluminum:21 atom %, a periodic table Ia, and an IIa group by 40 micrometers or less of boron:maximum droplet sizes - 6 atom % at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen, and a maximum droplet size is 50 micrometers or less, and it obtains the boride particle of 15 micrometers or less of mean particle diameters. Then, this maximum droplet size grinds a boride particle 50 micrometers or less, and considers as the boride particle of 5 micrometers or less of maximum droplet sizes.

[0025] The atmosphere in composition of a raw material and heat-treatment and temperature are the same as that of the case of the method which does not grind. Although the boride particle size compounded since the particle size of boron used as a raw material was coarse also becomes large here, even if a maximum droplet size uses a pulverization process by setting a mean particle diameter to 15 micrometers or less by 50 micrometers or less,-izing can be carried out [detailed] to 5 micrometers or less of maximum droplet sizes for a short time. Pulverization time can obtain boride powder in short ******** and the state where there is almost no mixing of an impurity. And a degree of sintering is also improved by using a boride particle with few impurities, and a precise sintered compact can be obtained.

[0026] It is desirable to perform pulverization by choosing conditions which an impurity does not mix as much as possible. As an example of a pulverization condition using the ball mill, product made from ball:alumina, diameter [of 3-6mm], and pulverization time: 4 - 10 hours are mentioned. As much as possible, grinding for a short time is desirable.

[0027] The detailed boride particle compounded as mentioned above can also obtain a sintered compact, using Ti, TiC, AlN, and aluminum2O3 grade as a binding material.

[0028]

[Embodiments of the Invention] Hereafter, the form of operation of this invention is explained.

(Example 1) the magnesium dust of 99.9% of purity, the amorphous boron powder of 1 micrometer or less of maximum droplet sizes, 99.9% of purity, the aluminium powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, and 180 micrometers of maximum droplet sizes -- respectively -- 85 atom % and ten atom % -- 5 atom % combination mixture was carried out This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid, and excessive aluminum was removed. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlMgB14 was shown. When the particle size analysis was performed, it was the detailed powder which consists of

particle size of 0.7 micrometers by 2 micrometers and average at the maximum.
[0029] After carrying out dryness processing of this powder at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. This Plastic solid was held at pressure 180MPa and the temperature of 1300 degrees C for 1 hour using hotpress equipment, and the sintered compact was obtained. The relative density to 2.6 g/cm3 of theoretical density was 98.6%, and when the obtained sintered compact measured Vickers hardness, it was 27GPa. When X diffraction measurement furthermore analyzed the sintered compact, the diffraction peak of MgAl 2O4 which is an oxide slightly was observed besides AlMgB14. From the calibration curve of the peak intensity ratio obtained from X diffraction measurement of the mixture whose composition is known, AlMgB14 was the content of 98.4 volume % in this sintered compact.
[0030] ~~(Example 2) The powder of AlMgB14~~ was produced like the example 1, and among the vacuum of 10-5torr (1.33x10-5kPa), and after carrying out dryness processing at 600 degrees C, pressing of this powder was carried out to the shape of a pellet. This Plastic solid was held for 30 minutes at pressure 2GPa and the temperature of 1300 degrees C using extra-high voltage elevated-temperature equipment, and the sintered compact was obtained. The obtained sintered compact was 32GPa, when the relative density to 2.6 g/cm3 of theoretical density has 99.7% of precise organization and measured Vickers hardness. As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 99.5 volume %.
[0031] (Example 3) the magnesium dust of 99.9% of purity, the crystalline-substance (trigonal system) boron powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, the aluminium powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, and 180 micrometers of maximum droplet sizes -- respectively -- 85 atom % and ten atom % -- 5 atom % combination mixture was carried out This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlMgB14 was shown. When the particle size analysis was performed, the maximum droplet size was 15 micrometers in the mean particle diameter at 50 micrometers or less.
[0032] Using the ball mill, this powder was ground until the maximum droplet size was set to 5 micrometers or less in ethanol. After carrying out dryness processing of the powder after pulverization at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. Using hotpress equipment, at pressure 180MPa and the temperature of 1200 degrees C, this Plastic solid was held for 1 hour, and was sintered. The obtained sintered compact was 97% of relative density to 2.6 g/cm3 of theoretical density, and Vickers hardness was 25.5GPa(s). As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 98.3 volume %.
[0033] (Example 4) Like the example 3, the pulverized-powder end of AlMgB14 was produced, and among the vacuum of 10-5torr (1.33x10-5kPa), and after carrying out dryness processing at 600 degrees C, pressing of this powder was carried out to the shape

of a pellet. This Plastic solid was held for 30 minutes at pressure 2GPa and the temperature of 1300 degrees C using extra-high voltage elevated-temperature equipment, and the sintered compact was obtained. The obtained sintered compact was 32.3GPa, when relative density has 99.5% of precise organization and measured Vickers hardness. As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 99.0 volume %.

[0034] (Example 5) Combination mixture of "99.9% of purity, the amorphous boron powder of 1 micrometer or less of maximum droplet sizes", and the "aluminum-28 atom % lithium alloy-powder end of 99.9% of purity and 300 micrometers or less of maximum droplet sizes" was carried out 30% of the weight with 70 % of the weight, respectively so that the composition ratio of boron, aluminum, and a lithium might become 82 atom %, 13 atom %, and 5 atom %. This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlLiB14 was shown. When the particle size analysis was performed, it was the detailed powder which consists of particle size of 0.8 micrometers by 3 micrometers and average at the maximum.

[0035] After carrying out dryness processing of this powder at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. This Plastic solid was held at pressure 180MPa and the temperature of 1300 degrees C for 1 hour using hotpress equipment, and the sintered compact was obtained. The obtained sintered compact was 98% of relative density to 2.46 g/cm3 of theoretical density, and when it measured Vickers hardness, it was 30GPa. As a result of performing X diffraction measurement like an example 1, the diffraction peak of AlLiO2 was observed as an oxide, and the content of AlLiB14 in a sintered compact was 99.6%.

[0036] (Example 1 of an examination) The sintered compact of examples 1-5 was joined on the base metal of cemented carbide, and periphery cutting of the round bar (phi150x300mm) of S45C (HRC35) which produced and heat-treated the cutting tool which made this the cutting edge was performed for 10 minutes. Cutting conditions are V=300 m/min, d= 0.5mm, f=0.15mm/rev., and dry type.

[0037] The cutting tool was prepared from the sintered compact which the maximum droplet size produced by the method of an example 3 held by the mean particle diameter by 50 micrometers or less as an example 1 of comparison, held the 15-micrometer particle at pressure 180MPa and the temperature of 1300 degrees C with the hotpress for 1 hour, and was obtained. Furthermore, the commercial alumina system ceramic tool and the cermet tool of marketing as an example 3 of comparison were also used as an example 2 of comparison. The result is shown in Table 2.

[0038]

[Table 2]

[0039] As shown in Table 2, the example showed the outstanding abrasion resistance and showed the longevity life. On the other hand, the sintered compact with coarse grain had low intensity, and suffered a loss in the middle of cutting. An alumina tool is in the middle of processing, and ~~showed the short life by the deficit~~. Toughness of the cermet tool was low and the edge of a blade damaged it completely simultaneously with a cutting start.
[0040] (Example 2 of an examination) The **-ed material which put four V character slots into the round bar (phi150x300mm) of SCN435 (HRC40) which produced and heat-treated the cutting tool of an example and the example of comparison like the example 1 of an examination was prepared, and intermittent cutting by which the load of the shock is carried out to a tool was performed. Cutting conditions are V=200 m/min, d= 0.2mm, f=0.12mm/rev., and dry type. The result is shown in Table 3.
[0041]
[Table 3]

[0042] As shown in Table 3, the example showed the outstanding deficit-proof nature. On the other hand, since intensity was insufficient for the sintered compact and the alumina tool with coarse grain, they suffered a loss in early stages of cutting. The heat check occurred and the cermet tool suffered a loss.
[0043]
[Effect of the Invention] It has the abrasion resistance and deficit-proof nature which were excellent when this invention sintered compact had the high content of a boride, it was constituted as a sintered compact which consists only of a boride substantially and it used as a cutting tool, as explained above.
[0044] Moreover, the manufacture method of this invention sintered compact can obtain the precise sintered compact of a high degree of hardness by using the raw material powder of a particle.

TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the suitable boride sintered compact for antifriction material or a cutting tool, and its manufacture method. Especially, it is related with the optimal boride sintered compact and its optimal manufacture method of a high degree of hardness for a processing abrasion-resistant tool or a cutting tool of steel or cast iron.

PRIOR ART

[Description of the Prior Art] Although aluminum 2O3 for cutting and Si3N4 ceramic tool are excellent in abrasion resistance, they are lacking in toughness, and they cannot acquire tool life sufficient in intermittent cutting. On the other hand, although a cBN sintered compact shows a long-life life more, the cBN powder raw material compounded under extra-high voltage is expensive. Furthermore, since a cBN sintered compact sinters by the extra-high voltage of 4 or more GPas which is the stable zone of cBN, even if a tool unit price is high and it long lasting, a cost merit cannot be obtained. [unit price]
[0003] On the other hand, the single crystal of AlMgB14 or AlLiB14 shows an about 3000-Hv high degree of hardness, and the dogged crystal structure can be expected as a tool material which combined deficit-proof nature and abrasion resistance.
[0004] As conventional technology about AlMgB14, it is W.Higashi. and T.Ito J.Less The technology of a publication is known by Comm.Met.92 (1983) 239. This paper dissolves magnesium and boron into a lot of aluminum melts, and the method of depositing a crystal is indicated.
[0005] Moreover, the technology of a publication is known by USP6099605 as another conventional technology. This official report shows that the sintered compact of a high degree of hardness is obtained by sintering the powder of aluminum, Mg, and B element with hotpress equipment, after turning minutely using the mechanical alloying method.

EFFECT OF THE INVENTION

[Effect of the Invention] It has the abrasion resistance and deficit-proof nature which were excellent when this invention sintered compact had the high content of a boride, it was constituted as a sintered compact which consists only of a boride substantially and it used as a cutting tool, as explained above.
[0044] Moreover, the manufacture method of this invention sintered compact can obtain the precise sintered compact of a high degree of hardness by using the raw material powder of a particle.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] By the method of the aforementioned paper, AlMgB14 particle dozens of micrometers or more is generable. More specifically, the

minimum is also 50 micrometers and particle size amounts to 2mm at the maximum. However, there was a problem that a precise sintered compact could not be obtained, in such coarse grain. Moreover, when such coarse grain is ground, detailed-ization takes a long time, an impurity mixes in 3 % of the weight or more and a large quantity from a pulverization container or tumbling medias (ball etc.), and the sintered compact which can evaluate abrasion resistance and deficit-proof nature is not obtained.
[0007] On the other hand, in order for the pulverization equipment of the high-energy type into which a steel crushing steel ball and steel raw material powder were put to perform mechanical alloying process to USP6099605 with the technology of a publication, impurity mixing of a crushing steel ball or the container quality of the material is not avoided. Therefore, it is checked that iron and raw material boron react, and FeB49 is generating and mixing. The 2 yuan boride of such a metal is a brittle material, and when a sintered compact is ~~manufactured and it uses~~ as a cutting tool, there is a problem referred to as reducing the deficit-proof nature of the edge of a blade. Moreover, since there is fear of explosion by still more rapid oxidation reaction, it is [that it is easy to produce oxidation reaction in mechanical alloying] substantially impossible to turn minutely the boride containing Ia(s), such as Li and Na, and an IIa group metal, and to obtain a sintered compact.
[0008] Therefore, the key objective of this invention is to offer a boride sintered compact equipped with deficit-proof nature required for cutting of steel etc., and its manufacture method. Moreover, other purposes of this invention are to offer the high boride sintered compact and its manufacture method of density by the low cost.

MEANS

[Means for Solving the Problem] this invention attains the above-mentioned purpose by specifying composition, the particle size of a raw material, or the synthetic conditions of raw material powder. That is, this invention boride sintered compact is a boride sintered compact containing the boride which consists of one element M chosen from aluminum, a periodic table Ia, and an IIa group metal, and boron. This boride sintered compact consists of the boride and unescapable impurity by which composition is expressed with AlX<=1My<=1BZ>=12. And it is characterized by the content of the boride occupied to this sintered compact being more than 98 volume %.
[0010] Conventionally, the sintered compact containing the boride to which composition is expressed with AlX<=1My<=1BZ>=12 has the low content of a boride, for example, the impurity is contained in USP6099605 to 10% of the weight of order with the technology of a publication. This is because an impurity mixes in that case since pulverization which destroys the crystal structure nearly completely by mechanical alloying is performed to raw material powder. In this invention, by using for a raw material the detailed powder with which the crystal structure has not collapsed as much as possible, the high sintered compact of the content of a boride can be obtained and the optimal high degree of hardness for a cutting tool and deficit-proof nature can be combined.
[0011] this invention sintered compact consists of a 3 yuan boride to which composition is expressed with AlX<=1My<=1BZ>=12, and an unescapable impurity of under 2

volume %. AlMgB14, AlLiB14, etc. are mentioned as composition of a more concrete boride. In AlMgB14, more exact structure is aluminum0.75Mg 0.78B14. As an unescapable impurity, generally, by the aluminum-Mg-B system, the oxide containing aluminum and Mg is seen and the oxide containing aluminum and Li is seen by the aluminum-Li-B system. As an oxide containing aluminum and Mg, AlLiO2 is mentioned as an oxide with which MgAl 2O4 contains aluminum and Li.

[0012] It is desirable to set to 3 micrometers or less the maximum droplet size of the boride which constitutes a sintered compact, and to set the mean particle diameter of a boride to 1 micrometer or less. By constituting such a detailed boride, the precise sintered compact of a high degree of hardness can be obtained. The degree of hardness of a sintered compact desirable as a cutting tool is more than Hv25GPa. Moreover, 97% or more of the relative density to the theoretical density of a sintered compact is desirable.

[0013] On the other hand, the manufacture method of this invention boride sintered compact is characterized by having the process at which a maximum droplet size sinters a boride particle (3 micrometers or less or 5 micrometers or less) less than [more than pressure:150MPa10GPa] and less than [more than temperature:1000 degree-C1500 degree C].

[0014] By using a detailed boride particle, the precise sintered compact of a high degree of hardness can be obtained. Such a boride particle is suitable as raw material powder at the time of being unable to break down the crystal structure as much as possible because it considers [whether it grinds and] as short-time pulverization, and being able to consider as a state with few impurities, and producing the high sintered compact of a boride content. The more desirable range of 1 or more-GPa 5 or less GPa of a sintering pressure is 3 or less GPa more than per GPa still more preferably. The more desirable range of sintering temperature is 1200-1400 degrees C. The sintering holding time has about 15 - 60 desirable minutes.

[0015] The detailed boride particle used as the raw material powder of a sintered compact has two kinds, the method of obtaining the boride particle of a particle from the beginning, without grinding, and the method of making it into a particle by obtaining coarse grain first and grinding after that.

[0016] The method which does not grind can be acquired according to the process which heat-treats the raw material which has composition of one element M:3 chosen from 75 to 91 atom %, four to aluminum:21 atom %, a periodic table Ia, and an IIa group by 1 micrometer or less of boron:maximum droplet sizes - 6 atom % at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen.

[0017] The maximum droplet size of the boride particle obtained can be held down to 3 micrometers or less because a maximum droplet size uses boron 1 micrometer or less as a raw material.

[0018] Conventionally, in AlMgB14, in order to carry out a crystal growth, many aluminum was contained. For example, the content of aluminum is a 80 - 91 atom % grade. In this invention, the content of aluminum was reduced as much as possible, and the composition from which a detailed boride particle is obtained was chosen. When it separates from the composition range specified above, a grain growth happens and there is an inclination used as the boride particle of coarse grain.

[0019] The example of composition from which the content of aluminum was changed and the boride particle of a particle was obtained by the above-mentioned method about

AlMgB14 is shown in Table 1. Some oxide was also seen, although composition No.1 in this table 1 is the stoichiometric composition of AlMgB14 and the detailed boride particle was obtained. Moreover, the obtained particle was a particle and No.2 were desirable AlMgB14 also with very little oxide. Furthermore, although No.3 have compounded AlMgB14 of a particle, it has coarse-grain-ized compared with No.2, and 21 atom % is thought as an amount of aluminum from which a desirable particle is obtained.
[0020]
[Table 1]

[0021] Atmosphere of heat-treatment is made into inert gas atmosphere. However, nitrogen is removed. Usually, it is suitable to use an argon.
[0022] Heat-treatment temperature should just be the temperature not more than temperature -1300 degree C beyond the melting point (660.4 degrees C) of aluminum theoretically. It is not desirable in order to compound AlB12 simultaneously, when it compounds at the temperature exceeding 1300 degrees C. 1200-1300 degrees C especially of optimal synthetic temperature are before and after 1200 degrees C. About 30 - 90 minutes of the holding time are desirable.
[0023] When it was this method, and the boride particle of a particle can be obtained, an impurity mixes at the time of pulverization and it considers as a sintered compact, without grinding, it can avoid that the content of a boride falls.
[0024] Next, the method of grinding heat-treats the raw material which has composition of one element M:3 first chosen from 75 to 91 atom %, four to aluminum:21 atom %, a periodic table Ia, and an IIa group by 40 micrometers or less of boron:maximum droplet sizes - 6 atom % at the temperature of 1300 degrees C or less in inert gas atmosphere other than nitrogen, and a maximum droplet size is 50 micrometers or less, and it obtains the boride particle of 15 micrometers or less of mean particle diameters. Then, this maximum droplet size grinds a boride particle 50 micrometers or less, and considers as the boride particle of 5 micrometers or less of maximum droplet sizes.
[0025] The atmosphere in composition of a raw material and heat-treatment and temperature are the same as that of the case of the method which does not grind. Although the boride particle size compounded since the particle size of boron used as a raw material was coarse also becomes large here, even if a maximum droplet size uses a pulverization process by setting a mean particle diameter to 15 micrometers or less by 50 micrometers or less,-izing can be carried out [detailed] to 5 micrometers or less of maximum droplet sizes for a short time. Pulverization time can obtain boride powder in short ******** and the state where there is almost no mixing of an impurity. And a degree of sintering is also improved by using a boride particle with few impurities, and a precise sintered compact can be obtained.
[0026] It is desirable to perform pulverization by choosing conditions which an impurity does not mix as much as possible. As an example of a trituration condition using the ball mill, product made from ball:alumina, diameter [of 3-6mm], and trituration time: 4 - 10 hours are mentioned. As much as possible, grinding for a short time is desirable.

[0027] The detailed boride particle compounded as mentioned above can also obtain a sintered compact, using Ti, TiC, AlN, and aluminum2O3 grade as a binding material.
[0028]
[Embodiments of the Invention] Hereafter, the gestalt of operation of this invention is explained.
(Example 1) the magnesium dust of 99.9% of purity, the amorphous boron powder of 1 micrometer or less of maximum droplet sizes, 99.9% of purity, the aluminium powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, and 180 micrometers of maximum droplet sizes -- respectively -- 85 atom % and ten atom % -- pentatomic % combination mixture was carried out This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid, and excessive aluminum was removed. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlMgB14 was shown. When the particle size analysis was performed, it was the detailed powder which consists of particle size of 0.7 micrometers by 2 micrometers and average at the maximum.
[0029] After carrying out dryness processing of this powder at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. This Plastic solid was held at pressure 180MPa and the temperature of 1300 degrees C for 1 hour using hotpress equipment, and the sintered compact was obtained. The relative density to 2.6 g/cm3 of theoretical density was 98.6%, and when the obtained sintered compact measured Vickers hardness, it was 27GPa. When X diffraction measurement furthermore analyzed the sintered compact, the diffraction peak of MgAl 2O4 which is an oxide slightly was observed besides AlMgB14. From the calibration curve of the peak intensity ratio obtained from X diffraction measurement of the mixture whose composition is known, AlMgB14 was the content of 98.4 volume % in this sintered compact.
[0030] (Example 2) The powder of AlMgB14 was produced like the example 1, and among the vacuum of 10-5torr (1.33x10-5kPa), and after carrying out dryness processing at 600 degrees C, pressing of this powder was carried out to the shape of a pellet. This Plastic solid was held for 30 minutes at pressure 2GPa and the temperature of 1300 degrees C using extra-high voltage elevated-temperature equipment, and the sintered compact was obtained. The obtained sintered compact was 32GPa, when the relative density to 2.6 g/cm3 of theoretical density has 99.7% of precise organization and measured Vickers hardness. As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 99.5 volume %.
[0031] (Example 3) the magnesium dust of 99.9% of purity, the crystalline-substance (trigonal system) boron powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, the aluminium powder of 40 micrometers or less of maximum droplet sizes, 99.9% of purity, and 180 micrometers of maximum droplet sizes -- respectively -- 85 atom % and ten atom % -- pentatomic % combination mixture was carried out This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high

grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlMgB14 was shown. When the particle size analysis was performed, the maximum droplet size was 15 micrometers in the mean particle diameter at 50 micrometers or less.

[0032] Using the ball mill, this powder was ground until the maximum droplet size was set to 5 micrometers or less in ethanol. After carrying out dryness processing of the powder after pulverization at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. Using hotpress equipment, at pressure 180MPa and the temperature of 1200 degrees C, this Plastic solid was held for 1 hour, and was sintered. The obtained sintered compact was 97% of relative density to 2.6 g/cm3 of theoretical density, and Vickers hardness was 25.5GPa(s). As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 98.3 volume %.

[0033] (Example 4) Like the example 3, the pulverized-powder end of AlMgB14 was produced, and among the vacuum of 10-5torr (1.33x10-5kPa), and after carrying out dryness processing at 600 degrees C, pressing of this powder was carried out to the shape of a pellet. This Plastic solid was held for 30 minutes at pressure 2GPa and the temperature of 1300 degrees C using extra-high voltage elevated-temperature equipment, and the sintered compact was obtained. The obtained sintered compact was 32.3GPa, when relative density has 99.5% of precise organization and measured Vickers hardness. As a result of performing X diffraction measurement like an example 1, the content of AlMgB14 was 99.0 volume %.

[0034] (Example 5) Combination mixture of "99.9% of purity, the amorphous boron powder of 1 micrometer or less of maximum droplet sizes", and the "aluminum-28 atom % lithium alloy-powder end of 99.9% of purity and 300 micrometers or less of maximum droplet sizes" was carried out 30% of the weight with 70 % of the weight, respectively so that the composition ratio of boron, aluminum, and a lithium might become 82 atom %, 13 atom %, and 5 atom %. This end of mixed powder was put into the high grade alumina crucible, and it installed into the elevated-temperature atmosphere heating furnace, and it cooled, holding argon gas atmosphere, after holding at 1200 degrees C for 1 hour, among 99.99% or more of high grade argon gas atmosphere, and. Dissolution processing of the powder after heating was carried out in the hydrochloric acid. When the powder which remained was investigated by X diffraction measurement, the diffraction peak of only AlLiB14 was shown. When the particle size analysis was performed, it was the detailed powder which consists of particle size of 0.8 micrometers by 3 micrometers and average at the maximum.

[0035] After carrying out dryness processing of this powder at 600 degrees C among the vacuum of 10-5torr (1.33x10-5kPa), pressing was carried out to the shape of a pellet. This Plastic solid was held at pressure 180MPa and the temperature of 1300 degrees C for 1 hour using hotpress equipment, and the sintered compact was obtained. The obtained sintered compact was 98% of relative density to 2.46 g/cm3 of theoretical density, and when it measured Vickers hardness, it was 30GPa. As a result of performing X diffraction measurement like an example 1, the diffraction peak of AlLiO2 was observed as an oxide, and the content of AlLiB14 in a sintered compact was 99.6%.

[0036] (Example 1 of an examination) The sintered compact of examples 1-5 was joined

on the base metal of cemented carbide, and periphery cutting of the round bar (phi150x300mm) of S45C (HRC35) which produced and heat-treated the cutting tool which made this the cutting edge was performed for 10 minutes. Cutting conditions are V=300 m/min, d= 0.5mm, f=0.15mm/rev., and dry type.

[0037] The cutting tool was prepared from the sintered compact which the maximum droplet size produced by the method of an example 3 held by the mean particle diameter by 50 micrometers or less as an example 1 of comparison, held the 15-micrometer particle at pressure 180MPa and the temperature of 1300 degrees C with the hotpress for 1 hour, and was obtained. Furthermore, the commercial alumina system ceramic tool and the cermet tool of marketing as an example 3 of comparison were also used as an example 2 of comparison. The result is shown in Table 2.

[0038]

[Table 2]

[0039] As shown in Table 2, the example showed the outstanding abrasion resistance and showed the long-life life. On the other hand, the sintered compact with coarse grain had low intensity, and suffered a loss in the middle of cutting. An alumina tool is in the middle of processing, and showed the short life by the deficit. Toughness of the cermet tool was low and the edge of a blade damaged it completely simultaneously with a cutting start.

[0040] (Example 2 of an examination) The **-ed material which put four V character slots into the round bar (phi150x300mm) of SCN435 (HRC40) which produced and heat-treated the cutting tool of an example and the example of comparison like the example 1 of an examination was prepared, and intermittent cutting by which the load of the shock is carried out to a tool was performed. Cutting conditions are V=200 m/min, d= 0.2mm, f=0.12mm/rev., and dry type. The result is shown in Table 3.

[0041]

[Table 3]

[0042] As shown in Table 3, the example showed the outstanding deficit-proof nature.

On the other hand, since intensity was insufficient for the sintered compact and the alumina tool with coarse grain, they suffered a loss in early stages of cutting. The heat check occurred and the cermet tool suffered a loss.